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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BAUXITE

PART 6 DETERMINATION OF VANADIUM

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BAUXITE

PART 6 DETERMINATION OF VANADIUM

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BAUXITE

PART 6 DETERMINATION OF VANADIUM

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 6) (First Revision) was adopted by the Indian Standards Institution on 24 December 1985, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 2000-1962* which covered determination of loss on ignition; total silica, non-reactive and reactive silica; alumina; ferric oxide by dichromate method, permanganate and colorimetric methods; titania by reduction (volumetric) and colorimetric methods; phosphorus; calcium oxide; magnesium oxide and manganous oxide was first published in 1962. While reviewing this standard the Sectional Committee felt the need for the revision of this standard. On the basis of experience gained during these years the Committee decided to revise this standard into different parts covering determination of each element in a separate part, which on publication would supersede the relavant clauses of IS : 2000-1962* dealing with that element. This Part covers determination of vanadium. The other parts of this revised standard are as follows:

Part 1 Determination of loss on ignition

Part 2 Determination of silica (as SiO_2, 0·5 to 10·0 percent)

Part 3 Determination of alumina

Part 4 Determination of ferric oxide

Part 5 Determination of titania (0·5 to 12 0 percent)

Part 7 Determination of phosphorus pentoxide ($P_2O_5 < 0·5$ percent)

Part 8 Determination of manganous oxide ($MnO < 0·5$ percent)

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*Methods of chemical analysis of bauxite.

The procedures for determination of CaO and MgO in bauxite are at present under investigation and will be given in subsequent parts.

0.3 In this revision determination of vanadium by spectrophotometric method has been incorporated.

0.4 In preparation of this revision due consideration has been given to the facilities available in the country to carry out such analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 6) covers the determination of vanadium (for $V<0 \cdot 1$ percent) in bauxite.

2. SAMPLING

2.1 Sample shall be drawn and prepared as per the method precribed in **2** of IS : 2000 (Part 1)-1985†.

3. GENERAL

3.1 Use of Filter Papers — In this standard, Whatman filter papers with their relative numbers have been prescribed since they are commonly used. However, any other suitable brand of filter paper of corresponding porosity and of equivalent quality may also be used.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5. DETERMINATION OF VANADIUM AS PHOSPHOTUNGSTO-VANADATE (SPECTROPHOTOMETRIC METHOD)

5.1 Outline of the Method — Sample is fused with sodium carbonate and vanadium is extracted with water as sodium vanadate and estimated as phosphotungsto-vanadate complex.

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*Rules for rounding off numerical values (*revised*).

†Methods of chemical analysis of bauxite: Part 1 Determination of loss on ignition (*first revision*).

5.2 Reagents

5.2.1 *Sodium Carbonate* — Solid.

5.2.2 *Sodium Carbonate Solution* (*Wash Liquid*) — One percent aqueous solution (*m/v*).

5.2.3 *Dilute Sulphuric Acid* — 4 N (*v/v*).

5.2.4 *Phosphoric Acid* — 1 : 2 (*v/v*).

5.2.5 *Sodium Tungstate Solution* — Dissolve 16·5 g of sodium tungstate ($Na_2WO_4.2H_2O$) in 100 ml of water.

5.2.6 *Standard Vanadium Solution* — Dissolve 1·44 g of ammonium vanadate in water in a one-litre volumetric flask and make up the volume up to the mark. The above solution is diluted further to give a solution containing 0·5 mg of vanadium/ml.

5.3 Procedure

5.3.1 Weigh out accurately 0·2 g of the bauxite sample (*see* **2.1**) in a platinium crucible. Mix it thoroughly with about 1·5 to 2·0 g of sodium carbonate with a glass rod and collect the particles adhering to the glass with a brush. Fuse the mass thoroughly over a mecker burner. Mix the molten mass in the platinum crucible by rotating it thoroughly and then keep it swirling in the platinum crucible for about 15 minutes. After the complete decomposition, rotate the molten mass to solidify along the sides of the platinum crucible. Cool, add water (20 ml) and heat carefully to boiling to loosen the cake. Transfer the cake to a small beaker, rinse the crucible. Heat on a steam bath and hasten the disintegration of the cake by crushing with a flattened glass rod. When the residue has entirely disintegrated (volume about 50 ml) filter it through a filter paper of Whatman No. 42 or its equivalent. Wash the residue with hot sodium carbonate solution (about six to seven washings after transferring the precipitate to the filter paper).

5.3.2 Neutralize the filtrate with sulphuric acid and stir thoroughly to liberate carbon dioxide. Add sulphuric acid to make the solution approximately 0·5 N. The volume of the solution should be around 40 ml. Add 1·0 ml of phosphoric acid and 2·0 ml of sodium tungstate solution. Leave it for 2 hours, and transfer to a 50-ml or 100-ml volumetric flask. Wash the beaker with water and make the volume up to the mark. Mix well before measurements. Determine the absorbance of a solution at 410 nm in a suitable spectrophotometer using 2 cm (for 50 ml volume) or 4 cm (for 100 ml volume) cell. A process blank should be carried out using chemical used and any value obtained thereof should be deducted.

5.3.3 Take 0, 1, 2, 3 and 4 ml of standard vanadium solution and add sulphuric acid to make the solution approximately 0·5 N and develop the colour following the procedure in **5.3.2**. Measure the absorbance at 410 nm. Draw a standard calibration curve. Read the concentration of the sample from the calibration curve.

5.4 Calculation — Calculate the mg of vanadium present in the sample solution from the curve:

$$\text{Vanadium, percent} = A \times 0{\cdot}5$$

where

A = mg of vanadium in sample solution (obtained from the calibration curve).

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